High precision radiocarbon dating of archaeological waterlogged wood: focusing on wooden poles forming circular structures at the Mawaki site

考古遺跡出土木材の高精度放射性炭素年代測定:

真脇遺跡出土環状木柱列の編年研究への応用

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A dissertation for the degree of Doctor of Science

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2011

Abstract

In the coastal region around Noto Peninsula, Japanese archipelago, wooden circular structures, which might be used for ritual ceremony in the Final Jomon period, were detected from archaeological sites. The unique circular structures indicate the existence of independent human community in this region. Through chronological study based on radiocarbon dating of wooden poles forming the circular structures, it was estimated that the circular structures were constructed from 1,125 to 400 cal BC approximately. However, the estimated construction period was calculated by using calibrated ages of radiocarbon dates, and precise estimation of the period is difficult, because calibration curve corresponding to the Final Jomon period (*ca.* 1300-500 cal BC) is fluctuating mightily. In order to enhance precision of calibrated dates and estimate the construction period more accurately, we dated wooden poles by wiggle matching. At the Mawaki archaeological site, Ishikawa prefecture, Japan, six circular structures (circles A, B, C, D, E, and F) were detected, and some wooden poles were watersoaked to preserve from drying. As samples for wiggle matching analysis, tree rings were collected from the wooden poles belonging to the circles A, D or E, and F. Radiocarbon dates of the annual rings were measured with accelerator mass spectrometry at Center for Chronological Research, Nagoya University, and data sets of the radiocarbon dates were fitted to the IntCal09 calibration curve by Bayesian wiggle matching with an OxCal calibration program (v4.1.6). The wiggle matching results were combined with calibrated ages obtained by previous studies, and the construction period of the wooden circular structures at the Mawaki site was estimated precisely as around 890-540 cal BC. This period belongs to the last half (900-500 cal BC) of the Final Jomon period. In addition, construction periods of circles A, D/E, and F were dated as around 820-770 cal BC, 770-540 cal BC, and 740-680 cal BC, respectively.

In this study, we conducted radiocarbon dating and wiggle matching of the wooden poles excavated only at the Mawaki site, because most of the wooden poles excavated at the other sites were already conserved with polyethylene glycol (PEG). PEG is useful substance among any conservation materials, but it will be contaminants

made of dead carbon for radiocarbon dating. Removal of PEG must be attempted to the conserved woods before radiocarbon dating. However, effective elimination methods of PEG from the conserved woods are not established yet. Therefore, we tested four treatment methods (AAA, AAA + hot water, AAA + acetone, AAA + benzene) of the conserved woods to remove PEG. Radiocarbon dates of the treated samples were measured and compared with the true radiocarbon ages of samples to estimate efficiency of the PEG elimination. As the results, all radiocarbon dates of the treated samples were older than the true radiocarbon ages, and it was revealed that any treatments could not eliminate PEG completely from the conserved woods. We also estimated the most effective elimination method of PEG through comparison of radiocarbon dates among the treated samples. Radiocarbon dates of samples treated by AAA + hot water method showed the nearest values to the true dates. Therefore, we speculated that the AAA + hot water method is the most effective treatment to eliminate PEG.

On the other hand, in order to detect remaning PEG in the conserved woods, the wood samples were analyzed with PyGC/MS. Comparing with chromatograms between pure PEG and pure wood, characteristic peaks (C-n series) were observed for pure PEG but not for pure wood. Conserved woods with 4% remaining PEG were also analyzed by PyGC/MS, and the peaks of C-n series were observed on chromatograms. Thus we concluded that PEG remaining in the conserved woods can be detected using PyGC/MS definitely, and the detection of PEG with PyGC/MS will be help to measure accurate radiocarbon dates of woods conserved with PEG.

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1. Introduction

Radiocarbon dating has evolved chronological studies in archaeology. Especially, dating of charred material on pottery with accelerator mass spectrometry (AMS) introduced numerical and precise dates to the Jomon period (the earliest postglacial period in Japan characterized by hunter-gatherer communities). This period can be divided into six sub-periods and each sub-period was also assigned its age by the AMS radiocarbon dating method. As the next steps, many archaeologists have considerable interests in the dates of events occurred in the period. However, normal radiocarbon dating is not suitable in resolution to figure out the dates of events because of radiocarbon calibration problem. Through the calibration, probability offered by radiocarbon dates may spread widely in the calendar age scale and then temporal resolution is degraded. This problem notably affects chronological study in the Final Jomon period which lasted only 800 years from *ca.* 1,300 to 500 cal BC (northern Japan area) in the whole Jomon period lasted for 10,000 years. The expanded probability density by calibration of a radiocarbon date is too large compared with the time interval of the Final Jomon period. Then, to determine precise dates of archaeological events in the Final Jomon period, we have to adopt more accurate dating methods. Wiggle matching is regarded as one of the more precise dating methods. Although this method requires multiple samples from a temporal sequence such as tree rings or lake sediments, it can more higher precise calendrical dates than normal calibration method. In this study, therefore, we employed the wiggle matching to get precise ages of wooden circular structures that were discovered at the Mawaki archaeological site, Ishikawa prefecture, Japan.

The wooden circular structure is a characteristic archaeological remain in the Final Jomon period at the coastal region around Noto Peninsula and indicates that a human group with characteristic culture inhabited in this region. Commonly archaeological remains in the Jomon period are assigned their ages based on chronology established by pottery typology or stratigraphy. For the wooden structures excavated at the Mawaki site, however, there is no acceptable information to estimate their construction period accurately. To determine the date (construction period) of the circular structure, radiocarbon dating of outermost part of rings collected from wooden poles constituting structures had been conducted in previous studies and its calibrated dates revealed the construction period approximately as 1125-395 cal BC. However, it seems that the estimated construction period expanded unduly by the effect that each calibrated date has broad probability distribution (max. 400 years). Compared with the time interval of the Final Jomon period (800 years), the construction period inferred from the probability is too wide. In order to estimate more precise construction period of the structures, application of wiggle matching analysis is required.

For the complete chronological analysis of the wooden circular structures at the Mawaki site, all wooden poles should be dated. However, the wooden poles excavated at about 20 years ago have been designated as important cultural properties, and most of wooden poles were previously conserved with polyethylene glycol (PEG). Since PEG is an organic compound produced from fossil fuel, PEG in the wooden poles will be contaminants for radiocarbon dating. Therefore, we must remove PEG completely, before radiocarbon dating and wiggle matching of the wooden poles preserved with PEG. However, effective elimination of PEG is still not established. To verify the possibility and establish suitable washing process, we tested subtraction of PEG from the conserved wooden poles.

2. Radiocarbon dating

Radiocarbon dating, which is one of radiometric dating methods, is originally established by Libby, W. F., and this method has been widely used for chronological studies in archaeology, geology, and paleoenvironmental science. In this chapter, we summarize the principle of radiocarbon dating and explain briefly $\delta^{13}C$ correction and accelerator mass spectrometry, that are important knowledge to measure accurate radiocarbon dates.

2. 1. Principle

In natural, there are three carbon isotopes (^{12}C, ^{13}C, ^{14}C). ^{12}C is the most abundant isotope in carbon, and exist stably. ^{13}C is also a stable carbon isotope. In contrast, ^{14}C or radiocarbon is unstable, *i.e.*, radioactive isotope. Natural abundance of ^{14}C is remarkably lower than those of ^{12}C and ^{13}C (Table 2.1), and $^{14}C/^{12}C$ in nature is of the order of 10^{-12}.

Table 2. 1. Carbon isotopes in nature.

Isotopes	Mass Number	Stability	Abundance Ratio [%]
^{12}C	12	Stable	98.89
^{13}C	13	Stable	1.11
^{14}C	14	Unstable	0.00000000010

^{14}C is constantly formed by the reaction of nitrogen 14 (^{14}N) with neutrons produced by cosmic ray in the upper atmosphere (Libby 1947). The nuclear reaction is described as

$$n + {}^{14}N \rightarrow {}^{14}C + p \tag{1.1}$$

where, *n* and *p* is neutron and proton, respectively. The produced ^{14}C is rapidly oxidized to $^{14}CO_2$ and spreads widely in the troposphere. ^{14}C is incorporated into living matter such as plant and animal via photosynthesis and the food-chain. The incorporation of ^{14}C is conducted by metabolism of living matter, and ^{14}C will be supplied continuously during their lifetime. On the other hand, ^{14}C changes to ^{14}N due to radioactive decay in the following reaction

$${}^{14}C \rightarrow {}^{14}N + \beta^- + \bar{\nu}_e \tag{1.2}$$

where β^- is beta particle and $\bar{\nu}_e$ is electron antineutrino. The number of decays per time is proportional to the current number of radioactive atoms. This is expressed by the following differential equation.

$$\frac{dN}{dt} = -\lambda N \tag{1.3}$$

Where *N* is the number of radioactive atoms and λ is a positive number called the decay constant. As the solution to Eq. (1.3), the number of radioactive atoms *N* at time *t* can be written in exponential function of time

$$N = N_0 e^{-\lambda t} \tag{1.4}$$

where N_0 is number of radioactive atoms at $t = 0$. In this case, N_0 is the initial number of ^{14}C atoms when the radioactive decay started. Equation (1.4) is solved as

$$t = -\frac{1}{\lambda} ln\left(\frac{N}{N_0}\right) \tag{1.5}$$

Production and decay of ^{14}C are repeated persistently, ^{14}C concentration comes to equilibrium in atmosphere and also in animal or plant. Therefore, for radiocarbon dating, the initial ratio of ^{14}C atoms to the sum of all other carbon atoms at the time of the organism's death and hence the time when the radioactive decay started, is approximately the same as that of the atmospheric CO_2.

The decay rate of radioisotope, the number of decay per unit time, is controlled by decay constant λ as described in Eq. (1.3) or half-life. The half-life ($T_{1/2}$) is calculated in the following way:

$$T_{1/2} = (ln2)\tau \tag{1.6}$$

where τ is the mean lifetime which is inverse number of decay constant λ. The half-life of ^{14}C had been measured and discussed in the previous studies. To determine a consensus value of half-life, Libby (1955) summarized the reported values from Jones (1949) and Miller *et al*. (1950), and weighted mean of these determinations, 5568 ± 30 years, was authorized as the half-life of ^{14}C. However, according to further measurements of the half-life, more accurate value, 5730 ± 40 years, was reported (Godwin 1962). The new half-life is more reliable now, but the Libby's half-life of 5568 years is used for avoiding any confusion in radiocarbon age.

2. 2. $\delta^{13}C$ correction

Through decay of ^{14}C, natural isotopic fractionation arise. Fractionation is the term used to describe the differential uptake of one isotope. While three carbon isotopes are chemically indistinguishable, lighter ^{12}C atoms are preferentially taken up before the ^{13}C atoms in biological pathways. Similarly, ^{13}C atoms are taken up more than ^{14}C. An assumption is that the fractionation of ^{14}C relative to ^{12}C is almost twice that of ^{13}C, reflecting the difference in mass number. Fractionation must be corrected for in order to make use of radiocarbon measurements as a chronometric tool for all parts of the biosphere. To correct the effects of isotopic fractionation, the fractionation is then corrected, and normalized to -25 per mil. $\delta^{13}C$ is calculated by the following formula:

$$\delta^{13}C = \left(\frac{^{13}C/^{12}C}{(^{13}C/^{12}C)_{PDB}} - 1 \right) \cdot 10^3 \quad per\ mil \tag{1.8}$$

Radiocarbon concentration in a sample is expressed in relative to the NBS oxalic acid standard (HOxII) normalized to $\delta^{13}C$ = -25 per mil (Olson, 1970).

3. 3. Accelerator mass spectrometry

There are two principal methods of measuring residual ^{14}C activity. One is counting of bata-ray emitted from radiocarbon such as proportional gas counters, liquid scintillation spectrometers. The other is accelerator mass spectrometry (AMS), which has shifted the analytical concept from counting of radiocarbon decay products to direct counting of radiocarbon atoms in a sample. As the half-life of radiocarbon is relatively long, the number of radiocarbon atoms present in a sample compared to the number of radiocarbon beta-decays observed during 1 day of counting is ~3×10^6, significantly favoring the use of AMS.

The long half-life of radiocarbon also implies that the number of radiocarbon atoms in a contemporary sample is very large, about 6×10^{10} per gram. This number is needed to support the beta-ray counting rate of 15 dpm, and so, to reach a counting accuracy of ±1%, or ±80 year, requires a beta-ray counting time of about 10 hr for a gram of carbon. However, a 16-μA ion source current of ^{12}C ions implies a ^{14}C ion counting rate for modern carbon of ~120 ions per second from only a 1mg sample of contemporary carbon (Stuiver 1978).

3. Radiocarbon calibration

3. 1. Variation of radiocarbon concentration

Radiocarbon atomes produced by cosmic rays in the upper atmosphere oxidized to $^{14}CO_2$. Produced $^{14}CO_2$ rapidly mixes with other CO_2 molecules in the troposphere. Carbon atomes including ^{14}C exchange with reactive carbon reservoirs of the oceans and biospheres. In the past, fluctuations in the atmospheric ^{14}C concentration have been largely produced by changes in the solar activity (Stuiver *et al.* 1980). On longer time scales, changes in the Earth's magnetic field intensity affected the ^{14}C content of the atmosphere, producing positive ^{14}C anomalies during intervals of weaker geomagnetic field (Kigoshi *et al.* 1966).

3. 2. Calibration curve

In the late 1950's and early 1960's, researcher found that 14C of known age tree rings fluctuated up to a maximum of ±5% over the last 1500 years. In addition to long term fluctuations, smaller 'wiggles' were identified by Hessel de Vries (1958). This suggested that there were temporal fluctuations in ^{14}C concentration and radiocarbon dates of historical materials should be calibrated. Radiocarbon dates of sequential dendrochronologically dated trees primarily of US bristlecone pine, German and Irish oak have been measured over the past several 10 years to produce a calendrical/ radiocarbon calibration curve.

At present, consensus calibration curve was extended back to 50,000 years ago (IntCal09 calibration curve). For 0-12.4 cal kyr BP (Before Present, 0 cal BP=AD 1950) (Stuiver *et al*. 1997), data sets used in IntCal09 are established from tree rings which have well-known felling dates dendrochronologically. These trees were collected in Germany, Ireland, and North America, and radiocarbon calibration by IntCal09 can only apply to Northern Hemisphere samples, because there is a measurable difference between ^{14}C activities of dendrochronologically dated trees between the two hemispheres due to the limited mixing of atmospheric air between them. In addition, application of IntCal09 is restricted to only terrestrial samples.

3. 3. Wiggle matching

Because of variations in atmospheric radiocarbon concentrations in the past, radiocarbon age usually differs from the "true" calendar age of sample. To estimate the calendar age, the calibration curve, which shows the dependence of radiocarbon versus calendar ages, is used. Variations in the past radiocarbon concentration can influence the precision of dating by the radiocarbon method. For a single sample, the precision of a calibrated date is usually worse than the error of radiocarbon age. Because, in those periods, the calibration curve has large wiggles and then a radiocarbon age corresponds to a few calendar age range. On the other hand, those wiggles can improve the precision age determination when we are dealing with a series of samples.

The wiggle-matching technique can be applied for dating of tree ring sequences and sequences of annually laminated lake sediments. The wiggle matching technique was described in detail by Pearson (1986). With this method, radiocarbon ages from one series (a flouting curve) were compared with the calibration curve, and it gives the best fit point. The quality of the fit is expressed by the mean-square difference between radiocarbon ages of samples and the ages derived from the calibration curve. In practice, this difference (SS) is plotted versus the age of series, and the lowest SS value is regarded as the most probable value (T). Pearson (1986) discussed also the question of uncertainty of such an estimate, and noticed that if T is true, the statistics n•SS (where n is the number of samples) has a X^2 distribution, and argued that the confidence intervals of X^2/n (for any given probability P) can be used directly to determine confidence interval of T. This procedure, however, sometimes leads to erroneous results if we want to derive the probability distribution of T. On the other hand, the Bayesian approach enables us to calculate directly the probability distribution of the age of series (Chisten *et al.* 1995; Goslar *et al.* 1998; Bronk Ramsey *et al.* 2001). This can then be used to calculate a range of most likely dates in a similar way to the probability method of radiocarbon calibration.

4. Wooden Circular Structures

4. 1. Overview

In the coastal region facing the sea of Japan around Noto Peninsula in Japanese archipelago, wooden circular structures have been excavated from some wetland archaeological sites (Hashimoto 1994; Kato 1994; Nakajima *et al*. 1994; Minami 1994; Nishino 1994) (Figure 4.1). The structures are composed of chestnut (*Castanea crenata*) poles (*ca.* 1 m in diameter) arranged in the perfect circles of about 7 m diameters. Each pole constructing the structures was detected in a pit belonging Jomon culture layer. The structures are like a pit dwelling (typical habitation of the Jomon period). However, in the structures, there was no fire place characterizing pit dwelling, and the poles are too big (pit dwelling use *ca.* 30 cm poles). Some archaeologists have conjectures that the structures were religious architecture or storage building for harvested foods (Hashimoto 1994), but the usage of the structures is not understood yet. The wooden circular structures are a mysterious archaeological remain in Japan.

At the Chikamori site, Kanazawa, Ishikawa prefecture, Japan, the first excavation of circular structures was reported in 1980 (Minami 1983) (Figure 4. 2). From 1982 to 1983, similar structures were recognized at the Mawaki site, Noto, Ishikawa prefecture (Kato 1986). At the Yonaizumi site and the Sakuramachi site, the structures were also discovered (Nishino 1989; Oono 2005). The structures have been excavated from only in this region, then we presume that the structures is evidence, indicating existence of a cultural community in this region. Archaeological analyses

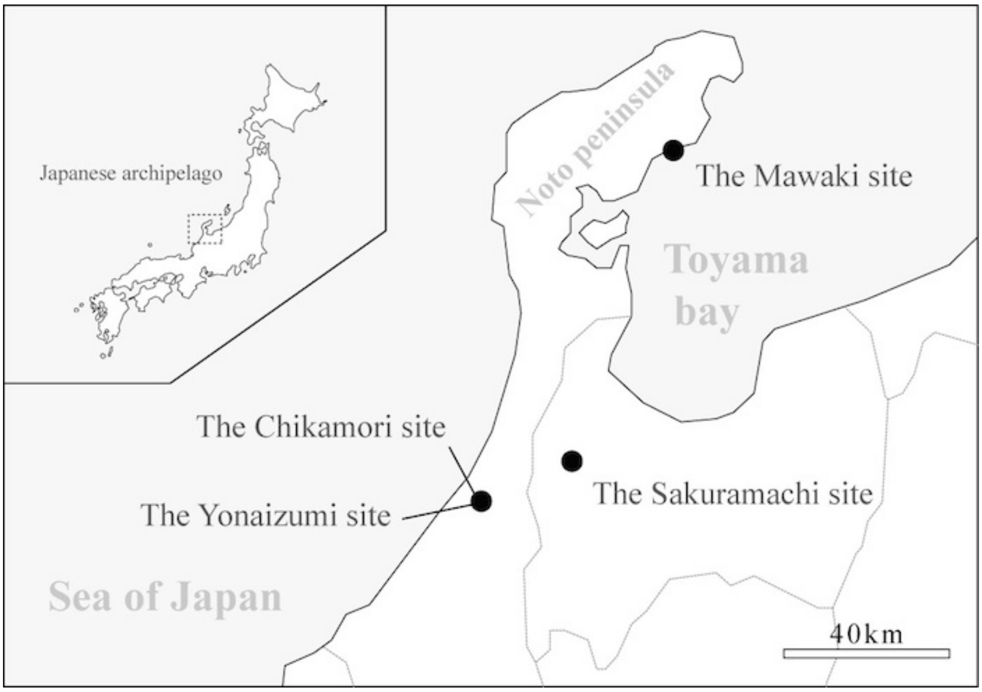

Figure 4. 1. Coastal region around Noto Peninsula. The archaeological sites excavated the wooden circular structures distribute only in this region.

such as stratigraphical and pottery-type studies dated the structures as the Final Jomon period, but they are inadequate to determine the precise duration of the structure because the Final Jomon period has a rather long time interval (*ca.* 1300-500 BC) (Oda and Yamamoto 2001; Kudo *et al*., 2008), precise dates in the Final Jomon period is necessary.

In order to determine the precise duration period of the structures, the radiocarbon dating had been attempted by previous studies. At the Sakuramachi site, three poles (SK186, SK126, SK158) constituting the wooden circular structures (circle A or B) were dated by radiocarbon dating method (Oono 2005) and the calibrated ages are distributed from *ca.* 1125 to 845 cal BC. These dates obtained are included within a time interval of the Final Jomon period, *i.e*., the results of radiocarbon dating are consistent with archaeological estimation. Also, at the Mawaki site, Yamada (1986) and Nakamura (2006) performed radiocarbon dating of outermost tree-rings picked up from each 16 poles constructing the structures (circle A, B, D, E and F), the calibration dates were calculated as *ca.* 895-395 cal BC. The calibrated dates can bring us interesting indication that there is time difference of the duration period of the structures between the Sakuramachi site and the Mawaki site. On the other hand, however, the calibrated dates are younger than the Final Jomon period. The disagreement between radiocarbon dating and archaeological estimation may arise from the radiocarbon calibration problem. If we can provide more precise dates, the disagreement might be eliminated.

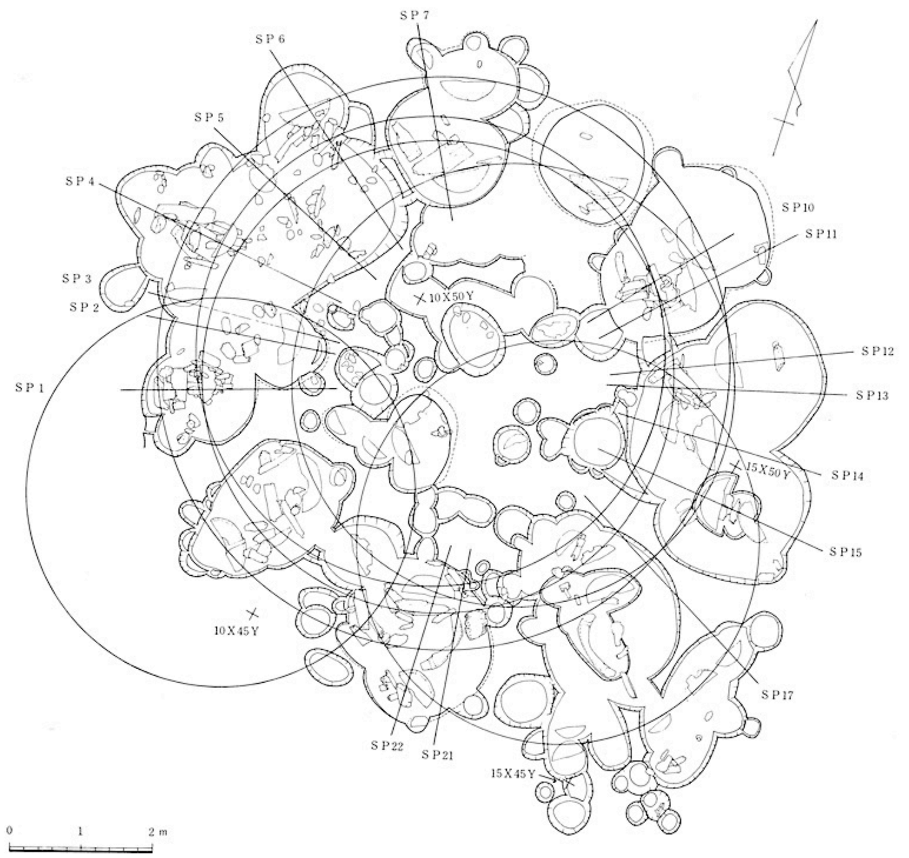

Figure 4. 2. Ground plan of the wooden circular structures excavated at the Chikamori site, Kanazawa, Ishikawa prefecture. Each circle means recognized structures (Kanazawa city 1983).

4. 2. The Mawaki archaeological site

The Mawaki archaeological site, one of the biggest archaeological sites in central Japan, is located on the alluvial coastal plain surrounded with hills. Excavation surveys revealed that the site was continuously occupied from the Early to Final Jomon period (*ca.* 5200 cal BC - 500 cal BC). For the wooden circular structures, totally 31 wooden poles have been excavated (Figure 4.3). The structures were detected for the first time during the excavation in 1982-83 (Kato *et al.*, 1986), and confirmed in 2002-2004 (Takada *et al.*, 2006). According to the archaeological studies, combinations of 6-10 poles among these 31 wooden poles formed 6 independent circular structures (named as circles A to F). All wooden poles are chestnut trees, being cut in half vertically and possessing no bark. The biggest pole belonging to circle A is about 1 m in

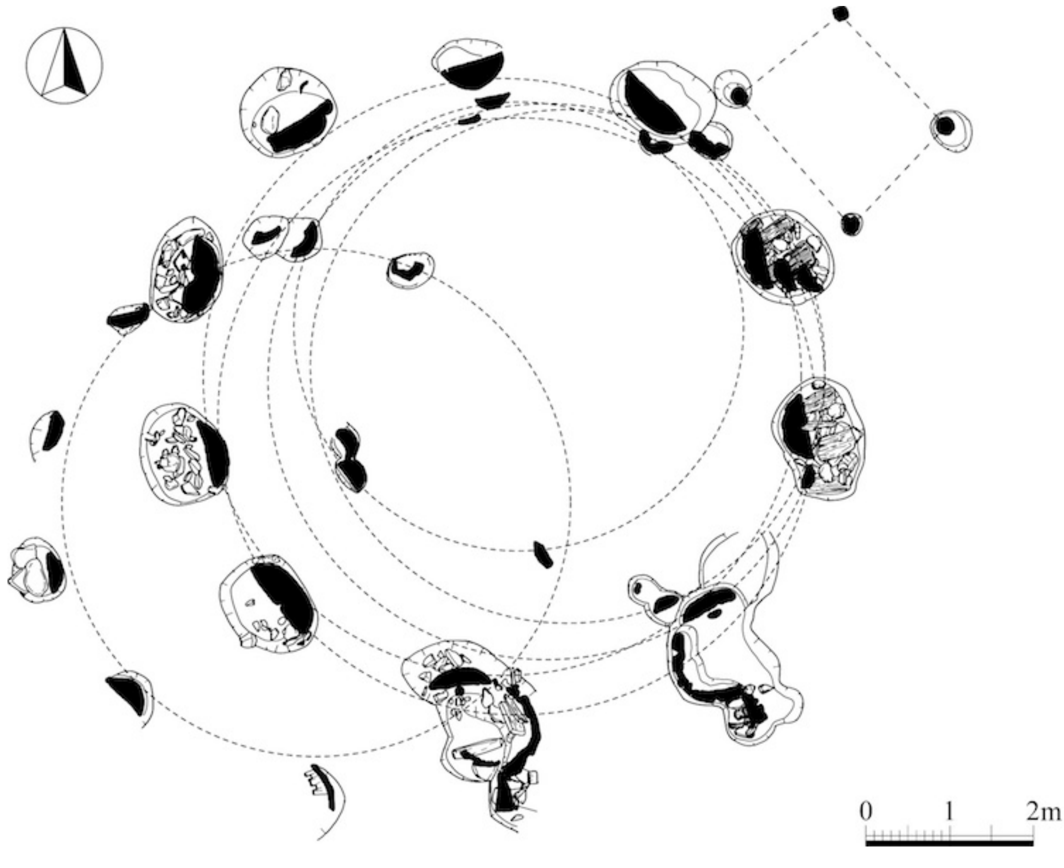

Figure 4. 3. Ground plan of the wooden circular structures excavated at the Mawaki site. Six circles show the structures recognized.

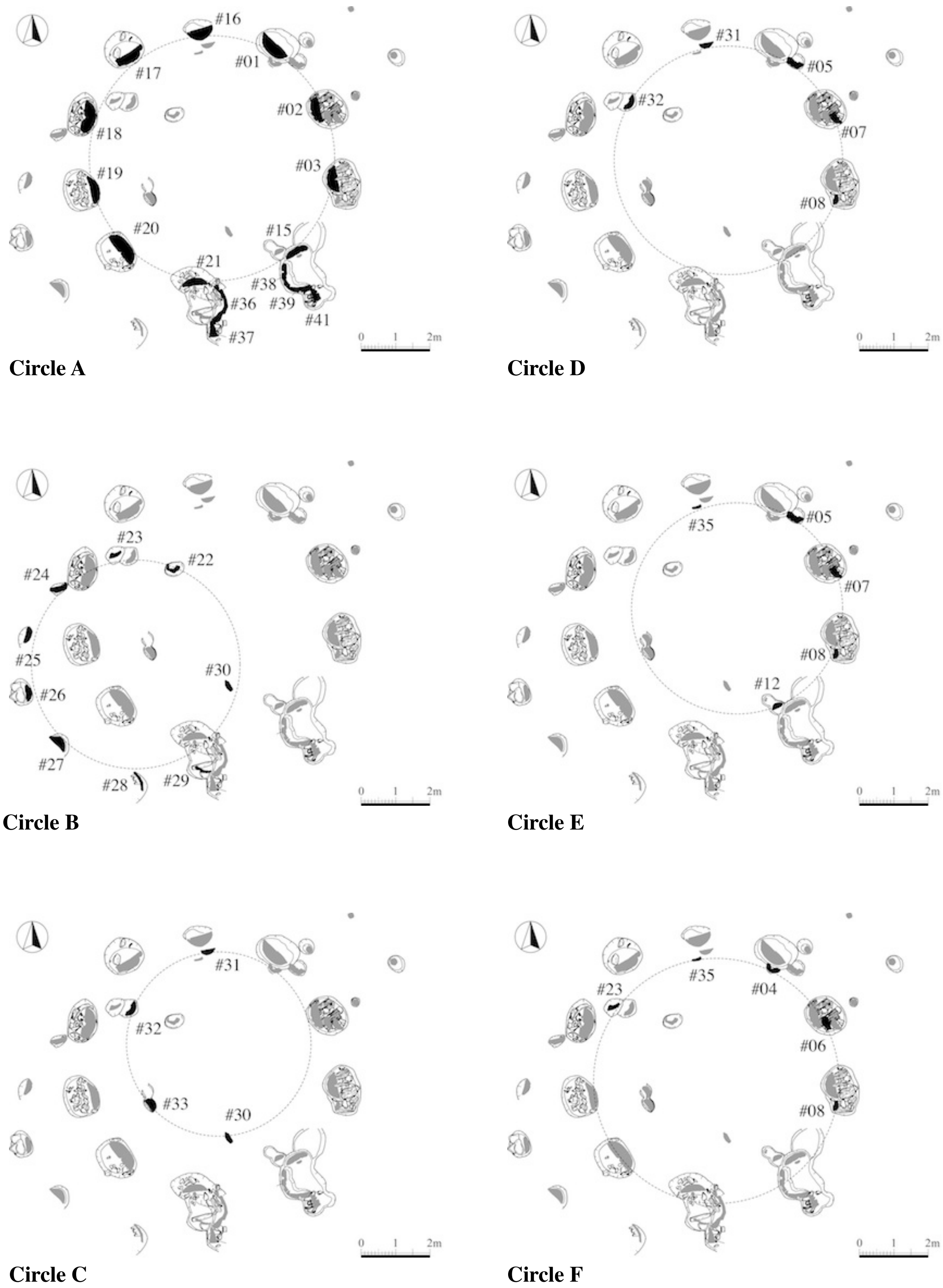

Figure4. 4. Wooden circular structures at the Mawaki site. Circle A and B have a gate-like structure. Some poles of each structure were diminished by degradation.

diameter, and 10 poles were used to make up the circle A. Each pole was set up in a pit of about 10-70 cm in depth from the ground level. Diameter of the circles is about 5-7 m, and circles A and B have a gate-like structure (Figure 4. 4). The six structures overlapped each other at nearby location indicating that these structures had been reconstructed five times consecutively. Among all archaeological sites, the Mawaki site has the most typical structures and the wooden poles are well preserved against weathering. Therefore, the circular structures at the Mawaki site are appropriate for chronological studies.

4. 3. Chronological studies

4. 3. 1. Archaeological approach

At the Mawaki site, a large number of pottery fragments were unearthed covering from the Early to Final Jomon period. Since a stratigraphic layer of the circular structures contains pottery fragments in the Final Jomon period, the dates of the structures were estimated as the Final Jomon period. Time interval of the Final Jomon period has been discussed according to the chronological studies using radiocarbon dating of charred material adhering on the pottery fragments. The Final Jomon period can be divided by pottery typology into four phases are Okyozuka, Nakaya, Shimono, and Nagatake types. Oda et. al. (2001) and Kudo et. al. (2008) conducted AMS radiocarbon dating of pottery fragments to these types. Consequently, it is estimated that the Final Jomon period lasted from 1300 BC to 500 BC approximately, and the circular structures at the Mawaki site are also dated within this period.

As mentioned above, the dates of the structures are estimated based on pottery typology. However, this layer contains also more older or newer pottery fragments during the Last Jomon period or the Early Yayoi period, respectively. Then, only from archaeological evidence, it is difficult to conclude the duration of the structure formation. In order to estimate more reliable and precise duration period, direct radiocarbon dating of wooden poles forming the circular structures was required as alternative approach.

Although the fundamental archaeological research could not date the duration period of the circular structures adequately, it can provide other chronological information. From the stratigraphical analysis at the Mawaki site, relative temporal sequences were well established between some poles, as poles from older to names: #01 (circle A) > #04 (F), #05 (D, E), #15 (A) > #12 (E), #02 (A) > #06 (F), #07 (D, E) (Appendix). This relationship indicates that circle A is older than circles D, E, F, and will contribute to confirm the consistency of radiocarbon dating results or wiggle matching analyses.

4. 3. 2. Radiocarbon dating

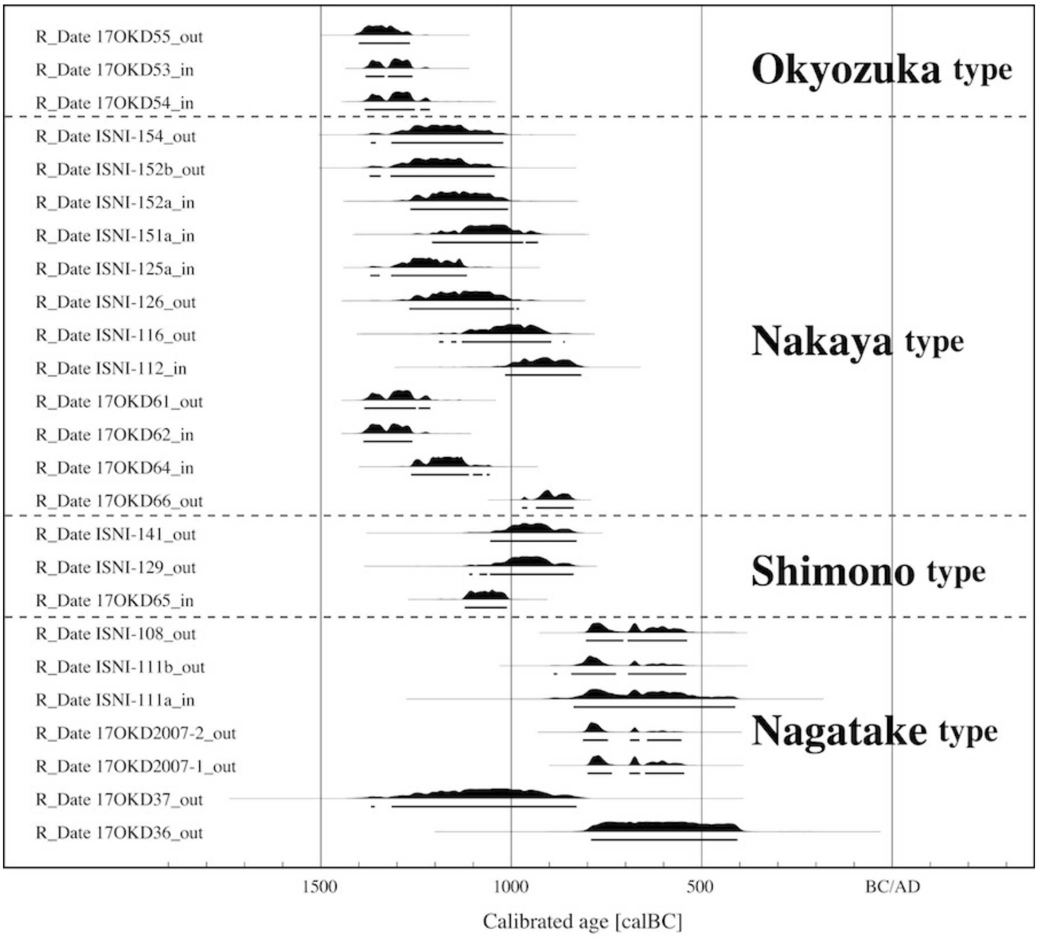

Figure 4. 5. Calibrated ages of charred materials on pottery of the Final Jomon period (Oda et. al. 2001; Kudo et. al. 2008).

In the previous studies, outermost rings picked up from the wooden poles constituting the wooden circular structures at the Sakuramachi site and the Mawaki site were dated by radiocarbon dating (Oono 2005; Yamada 1986; Nakamura 2006). As the result, calibrated radiocarbon dates were widely expanded from 1125-395 cal BC (Figure 4. 6). The radiocarbon dates indicate the possible period numerically, but they do not possess acceptable precision to discuss the duration of the structure formation. For example, at the Mawaki site, radiocarbon age for pole #02 (A) 2518 ± 34 BP. On the other side, the calibrated date for the pole has extremely wide age range over 200 years. Even for precise age determination, there is a limit to conventional radiocarbon calibration. In order to introduce more precise dates to the structures, we need to employ wiggle matching which is one of radiocarbon calibration techniques. Although wiggle matching necessitates many radiocarbon dates for ring samples from a tree ring sequence, felling dates for the poles can be dated precisely in calendrical ages.

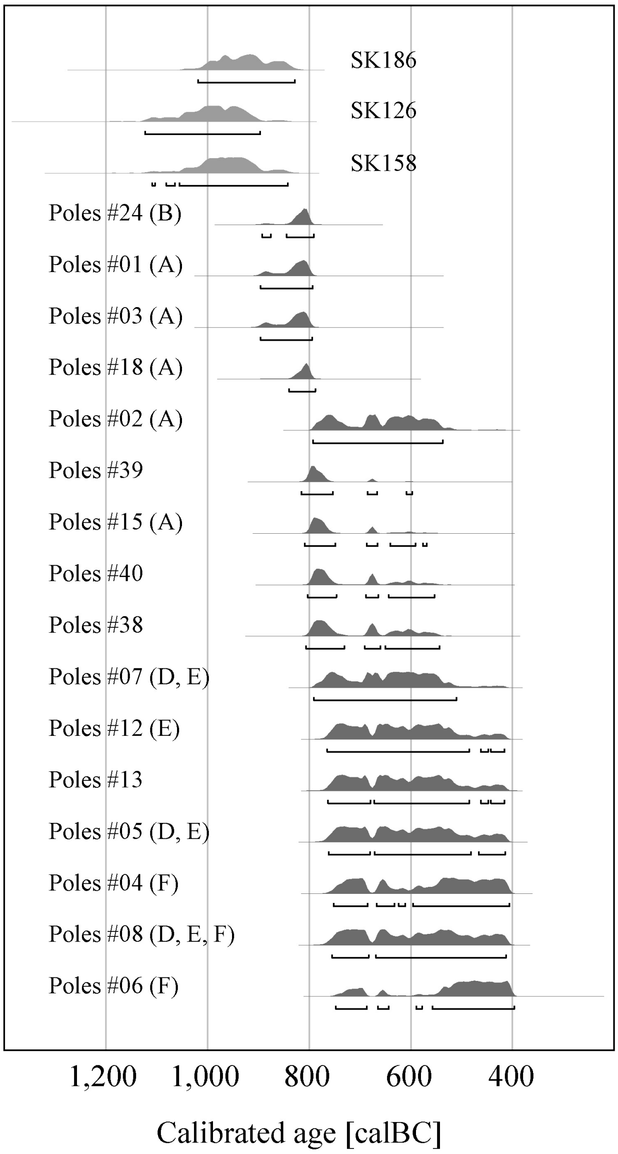

Figure 4. 6. Calibrated ages of outermost rings sampled from wooden poles constituting the wooden circular structures at the Sakuramachi and Mawaki sites.

4. 3. 3. Conservation

Waterlogged woods excavated from wetland sites were often degraded by microorganisms in the sediments. The microorganisms decompose cell walls which construct main structure of wood. When the woods were unearthed, the shape and size of the woods will be kept originally for a short period. However, time goes on, the woods will shrink abnormally. This dimensional change is induced by removal of surface tension of water in the woods. To conserve the waterlogged wood in original shape and size, we require to remove or replace carefully the inner water fraction with a chemical substance which can exist stably in the wood. As the impregnation substance, polyethylene glycol (PEG), in the following chemical formula,

$$HO - (CH_2 - CH_2 - O)_n - H$$

is widely employed in a great number of conservation laboratories (Grattan 1987; Rowell *et al*., 1990). Also wooden poles excavated in 1982-83 at the Mawaki site have been preserved with PEG. PEG is one of organic compounds and is commonly synthesized from fossil fuel. Therefore, the woods treated with PEG should involve dead carbon partly. When radiocarbon dates of the woods conserved with PEG were measured directly, the dates will be affected by dead carbon and shift to older age than true age. To determine accurate radiocarbon dates of waterlogged woods preserved with PEG, we must remove impregnated PEG from woods. In order to reveal suitable treatment processes for radiocarbon dating of chemically preserved woods, Bruhn *et. al.* (2001) tested radiocarbon dating of the conserved wood by removing PEG with several kinds of treatment including AAA (see, page 22). As the result, they implied that PEG can be removed with only standard AAA process. However, their experiments were attempted to only oak wood and the PEG soaking process into the wood is unknown. These attributions will affect the removal efficiency of PEG. To establish reliable methodology, experimental study containing above information is required for radiocarbon dating of wood conserved with PEG.

To establish absolute chronology by radiocarbon dating and wiggle matching, we want to measure all wooden poles constructing the wooden circular structures st the Mawaki site. However, most of the wooden poles excavated from the site were already preserved with PEG. Therefore, we should also investigate how to remove PEG from the conserved woods.

4. 3. 4. Dendrochronology

Radiocarbon dating and wiggle matching can determine the felling dates of wooden poles forming wooden circular structures precisely, but it is so hard for measurement of radiocarbon dates for all poles because wiggle matching analysis often require ten or more radiocarbon dates for each pole. To introduce felling dates more efficiently to the poles, dendrochronological approach seems to be effective. Dendrochronology is a dating method based on pattern matching of tree ring width variations among wooden samples. In Europe, Friedrich et. al. (2004) had established a 12,460 year-long absolute chronology combining Hohenheim oak and pine chronology. In previous study, dendrochronology was already conducted to the poles at the Mawaki archaeological site (Mitsutani 1986), but the felling dates can not be determined due to species of the wood (chestnut tree). The floating chronology is dated on calendrical time scale by comparison with the master chronology. In Japan, the master chronology for chestnut tree was not yet established, and hence dendrochronology is not available for dating of the wooden circular structures.

Here, if we compare some floating curves each other (cross-dating), it will provide relative chronology between each pole. Kimura et. al. (2004) had conducted cross dating mainly at the Aota site, Niigata prefecture, Japan. Theirs attempt indicated that the cross dating of broad leaf tree is possible under the right circumstances.

Although cross-dated woods have not calendar dates, it seems that relative chronology can be used for confirmation of consistency of wiggle matched dates. Ring width of the wooden poles was already measured and cross dated by K. Kimura, Fukushima University, relative chronology had been established at the Mawaki site. To verify wiggle matching results, comparison between wiggle matching and cross dating is required.

5. Experiment and analysis

To estimate construction period of the wooden circular structures, we conducted mainly radiocarbon dating and wiggle matching analyses of wooden poles constituting the circular structures at the Mawaki site. In addition, the poles were analysed dendrochronologically and some poles were cross-dated each other. However, unfortunately some wooden poles were already conserved with PEG, accurate radiocarbon dates of the conserved poles could not be obtained even without a careful treatment for elimination of dead carbon contamination.

5. 1. Radiocarbon dating and wiggle matching

In order to estimate the construction period based on wiggle matching, annual ring samples were picked up from nine poles comprising circles A, D, E and F (Table 5.1). The biggest pole (#01 or #03) has a large number of annual rings (over one hundred), but smaller poles have only few tens of annual rings. Firstly, wooden poles were snicked by using a disc saw, and a wooden block including full annual rings were separated from the poles. Totally, approximately 10 annual rings were collected from each pole.

Table 5. 1. Wooden poles and tree rings for wiggle matching analysis.

Pole No.	Circle	Pole Width [cm]	Number of Annual rings	Average Width of Annual Rings [mm]	Number of Measured Rings
01	A	93.6	132	3.07	11
02	A	73.0	50	5.82	10
03	A	72.4	119	2.29	6
15	A	71.0	32	6.26	8
05	D, E	50.0	40	4.13	11
07	D, E	40.0	23	3.46	13
12	E	34.5	42	3.29	12
04	F	41.0	89	1.83	13
06	F	39.0	51	2.59	13

5. 1. 1. Sample preparation

For radiocarbon dating with AMS, graphite targets prepared from tree ring samples are required. Here, we described the preparation processes of the tree rings.

5. 1. 1. 1 Acid-Alkali-Acid treatment

The tree ring samples may contain carbonate, humic acid, and other carbonaceous materials as potential contaminants for radiocarbon dating. Soil carbonate, which has old formation age, provides "old carbon" to a dated sample, and so the measured age shows apparently older than the original age of the sample, because contaminative ^{14}C-free carbonate decreases ^{14}C ratio of the dated sample. Humic acid in soil is formed under various conditions of burial environment as humidity, pH and earthiness. ^{14}C ratios of humic acid appear various values dependent on each formation age. Thus, existence of humic acid in dating sample effects problematic uncertainty to radiocarbon age. Fortunately these contaminants can be easily removed by acid and alkali solutions completely. Acid and alkali pretreatments can be applied to various materials as a conventional method for radiocarbon dating. In practice, hydrochloric acid solution (HCl) and sodium hydroxide solution (NaOH) are used as acid treatment and as alkali treatment, respectably. First of all, HCl treatment is performed to remove carbonate contaminants, and then NaOH treatment is carried out for removal of humic acid contaminants. At the end, second HCl treatment is needed because NaOH fraction should be extracted from the sample entirely. NaOH absorbs CO_2 and nonvolatiles. If a sample with residual NaOH is dated, obtained age may be younger depending on the amount of absorbed “modern carbon”. This procedure is called Acid-Alkali-Acid (AAA) treatment after its protocol. AAA treatment can be normally applied to charred materials, plants, paper, cloth and hairs. In this study, all wood samples were processed by AAA treatment. The sample was cleaned with an ultrasonic cleaner in distilled water 2 or 3 times for removal of soil detritus prior to the AAA treatment. After the cleaning, first HCl treatment was performed using 1.2 M HCl at 80°C overnight. Though NaOH treatment was also preformed under the same conditions as the HCl treatment eventually, initial addition of NaOH was begun from lower concentration of around 0.1M solution and no heating. Incompletely carbonized or inferiorly preserved charcoal damaged, *i.e.*, dissolved seriously by high concentration NaOH solution, because Na+ ion comes into interlaminar of graphite and the graphite layers slip each other. Al wood samples in NaOH solution elute dark brown fraction containing humic acid. This elution provides a good indication of the removal reaction. When the elution has stopped, the NaOH concentration and heating temperature are increased in small steps until 1.2 M and 80 °C. From samples with no elution, NaOH pretreatment carried out in the final conditions overnight. Second HCl treatment was preformed at 80℃ for 6 hours. Each sample was rinsed in distilled water and dried completely.

5. 1. 1. 2. CO_2 production

In order to obtain a pure graphite target for ^{14}C measurement, purification of CO_2 gas converted from a sample is needed. To gain CO_2 gas, first of all, a treated sample is combusted, and then CO_2 gas is separated from the produced gas by using refrigerants such as liquid nitrogen, cooled ethanol and n-pentane in a vacuum glass line.

5. 1. 1. 3. Graphite production

Reducing CO_2 gas, we can obtain graphite. The reduction process is fundamentally classified into two ways. The most common graphitization for radiocarbon dating is the reaction using H_2. Jull *et al*. (1986) showed details of the reaction. In CCR, this graphitization procedure has been applied for the first time as described in Kitagawa *et al*. (1993). The treated CO_2 and H_2 with iron (Fe) powder catalyist are sealed into a vycor tube. The amount of Fe powder is twice the carbon amount of each sample. Heating the sample tube at 650°C for 6h, graphite is produced on iron powder.

5. 1. 2. AMS radiocarbon measurement

^{14}C measurements of graphite targets that are prepared according to the above preparation processes have been conducted with an AMS system (High Voltage Engineering Europa Model-4130) at the Center for Chronological Research (CCR), Nagoya University. Radiocarbon dates were calculated using Libby's half life (5568 ± 30 years). $\delta^{13}C$ for collection of isotope fractionation were simultaneously measured along with ^{14}C concentration by AMS.

5. 1. 3. Wiggle matching

Wiggle matching in the simplest numerical approach is performed by using chi-square test (Pearson 1986). Although this method can estimate best fitting point on calibration curve, some assumptions are needed to define uncertainties (Bronk Ramsey *et al*, 2001). As a more advanced and strict statistical approach, Bayesian wiggle matching has been developed (Christen *et al*. 1995; Goslar *et al*. 2001) and employed in OxCal program (Bronk Ramsey 1995, 1998). The Bayesian process can provide efficiently probability distribution of calendar age of the outermost ring of a sample tree. To obtain high precision date of the poles and to analyze the introduced date more statistically, we applied the Bayesian method and used the OxCal program for wiggle matching.

5. 2. PEG elimination from conserved wood

To verify possibility of PEG elimination, we tested chemical removal of PEG from conserved wooden samples and radiocarbon dates of the washed samples were compared with true ages for each sample. However, if we measure radiocarbon dates of conserved woods practically, we should know whether impregnated PEG is completely removed or still remaining in each sample. To estimate degree of PEG elimination, the PEG concentration measurements with GC/MS were tested for wood samples.

5. 2. 1. Impregnation of PEG

For test sample of PEG elimination, we created wooden blocks from chestnut timbers excavated at the Sakuramachi site. The chestnut timbers were cut by a hand saw

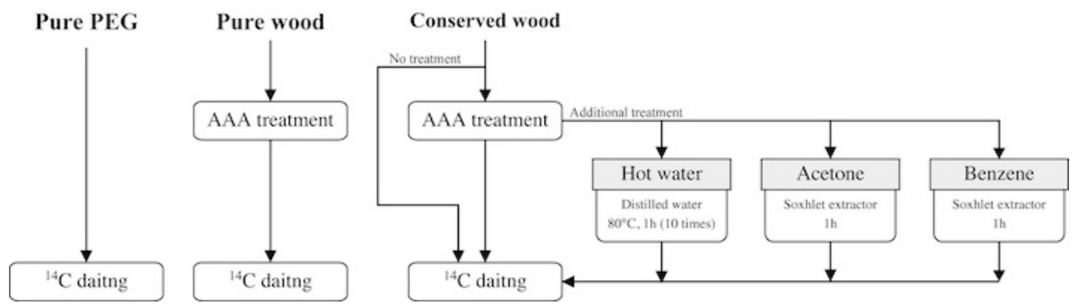

Figure 5. 1. Flowchart of PEG elimination process.

and a power saw, and the block with 30×30×15mm^3 which have similar annual rings were prepared from sapwood of the timbers. These blocks (named as SS series) were divided into two groups. One is for PEG impregnation (group 1) and the other is for no impregnation (group 2).

When the PEG material with same molecular weight is consistently used for the PEG impregnation process, the infiltration degree of PEG into the wooden blocks is deeply affected by PEG concentration in the water solution due to osmotic pressure. To infiltrate the PEG solution certainly, we started the soaking process from lower PEG concentration. Blocks belonging to group 1 were soaked in PEG (PEG4000s made by Sanyo Chemical Industries, Ltd.) water solution (20%) in a temperature controlled bath (60°C). After 2 weeks from the beginning, we changed the PEG water solution from 20% to 40% concentration. Continuously, PEG water solution was changed to more higher concentration per 2 weeks gradually and finally the concentration reached 100%. Totally we spent over six months for entire PEG impregnation processes. After impregnation, soaked blocks (group 1) were taken out from PEG solution and dried under room temperature.

As the other sample, we also got preserved wooden pieces from the Mawaki site. These pieces (named as Ar04P series) were a part of wooden poles constituting circle A that were excavated in 1982-83. They were already preserved with PEG in 1990s. It is supposed that there are some differences in PEG elimination efficiency between wood samples newly impregnated (group 1) and a few decades before. Thus these samples were also allotted for the elimination treatments. However, here we cannot know the true age of the wood pieces. Thus quantitative estimation of the elimination effect based on comparison of radiocarbon dates between treated wood and non-treated (pure) wood is impossible. To evaluate the elimination efficiency properly, true age of the wood sample is require. In this study, we estimated the true age of Ar04P series by dendrochronological cross-dating with wooden poles #01 which will be dated precisely by wiggle matching.

5. 2. 2. PEG elimination

In order to eliminate PEG from the conserved wood blocks, we tried not only normal AAA treatment, but also additional washing process using three chemical solvents, *i.e.*, hot water, acetone, and benzene. Firstly, $1\times1\times15mm^3$ pieces of wood samples were cut out from group 1 (SS series) and Ar04P series and the pieces were washed by AAA treatment.

After standard AAA treatment, there are three ways of methods for PEG elimination from the soaked blocks. One is washing with hot water in a beaker on a heating plate (80°C) for several times. It seems promising though the method is so simple or shoddy, because water has the highest solubility for PEG. The other two ways, acetone and benzene were employed independently for PEG solvents, and washing process were performed with a Soxhlet extractor which can persistently wash the sample through the distillation process. We operated the Soxhlet extractor for 1 hour for each solvent.

Washed samples were prepared to graphite target for radiocarbon dating. On the other hand, pure PEG, pure woods (SS series group 2) and conserved woods (SS series group 1 and Ar04P series) were prepared to graphite target without AAA or additional washing processes to evaluate contamination effect of PEG to the woods. All graphite targets were measured by AMS at CCR and radiocarbon ages were calculated.

5. 2. 3. Evaluation of PEG isolation by GC/MS

To estimate degree of PEG elimination from woods which were soaked in PEG solution in this study, we were able to use known age samples. In most cases, however, we can not obtain the true age of the samples. Therefore, we also need to know whether PEG was clearly removed from preserved wood or not before ^{14}C measurements. In order to evaluate the degree of PEG removal, PEG concentration analyses with GC/MS were conducted.

The Gas Chromatography/Mass Spectrometry (GC/MS) instrument separates chemical mixtures (the GC components) and identifies the components at a molecular level (the MS component). It is one of the most accurate tools for analyzing environmental samples. The GC works on the principle that a mixture will separate into individual substances when heated. The heated gases are carried through a column with an inert gas (such as helium). As the separated substances emerge from the column opening, they flow into the MS. The mass spectrometer identifies compounds by the mass of the molecular fragments. A “library” of known mass spectra, covering several thousand compounds, is stored on a computer. The mass spectrometer is considered as the definitive analytical detector.

In this study, we employed pyrolysis GC/MS (PyGC/MS) which can measure solid samples by quick pyrolysis to analyze wood samples. To uniform the sample condition, we milled woods by a mortar, and *ca.* 15mg milled wood samples were prepared for the PyGC/MS analysis.

6. Results

6. 1. Wiggle matching results

6. 1. 1. Circle A

From circle A, four poles (poles #01, #02, #03, #15) were dated by AMS radiocarbon dating and wiggle matching. Radiocarbon date of MWK15-28 (pole #15) showed an abnormal value owing to bad graphite target condition, so it was omitted for wiggle matching analysis of the poles. All results for radiocarbon dating were listed in each table (Appendix I). The radiocarbon ages were distributed in the range from *ca.* 2900 to 2500 BP which corresponded to a part of calibration curve forming a monotonic line. Therefore, all poles were dated precisely. Typical example is pole #02. The wiggle matching result is 790-770 cal BC, and it covers only 20 years in 95.4% probability. To demonstrate that wiggle matching is quite effective for highly precise dating, we tested fundamental calibration for an outermost ring of the pole #02. The result extremely expand the possible age range to 895 to 670 cal BC and it involves two ranges in the probability distribution (*i.e.*, 895-760 and 685-670 cal BC). Then we can conclude that highly precise dating was achieved successfully with wiggle matching analysis for the circle A. Poles #01, #03, and #15 were also dated as 820-790, 950-840, and 820-790 cal BC, respectively, and each date except for #03 belongs to a common age region of around 800 cal BC.

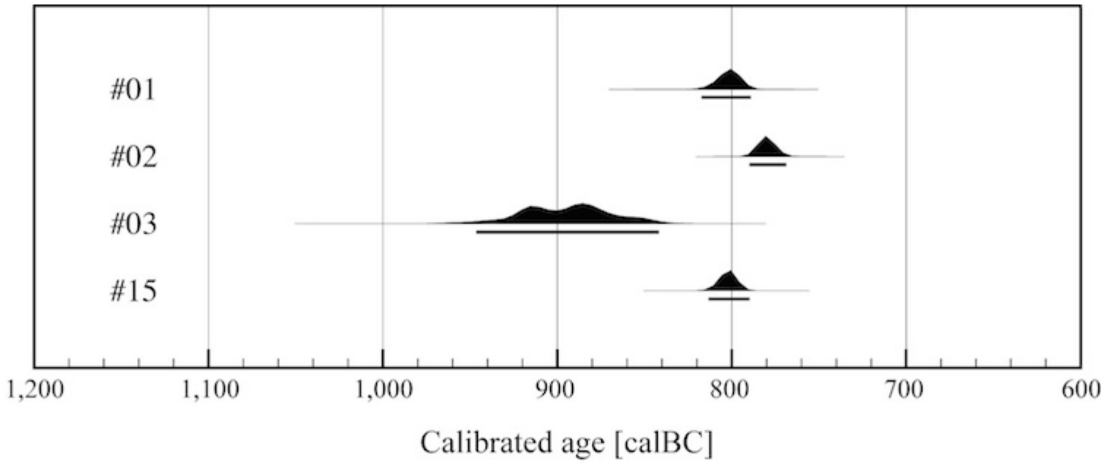

Figure 6. 1. Wiggle matching results for poles #01, #02, #03, and #15 belonging to circle A. Black lines under probability densities indicate the possible age ranges with 95.4% probability.

6. 1. 2. Circle D, E

We measured radiocarbon dates for poles #05, #07, and #12 forming circle D and/or E. The dates spread over around 2600-2450 BP which corresponds to mightily fluctuating part of the calibration curve. Then wiggle matching results for these radiocarbon dates yielded two or three possible age ranges for 95.4% probability (Figure 6. 2).

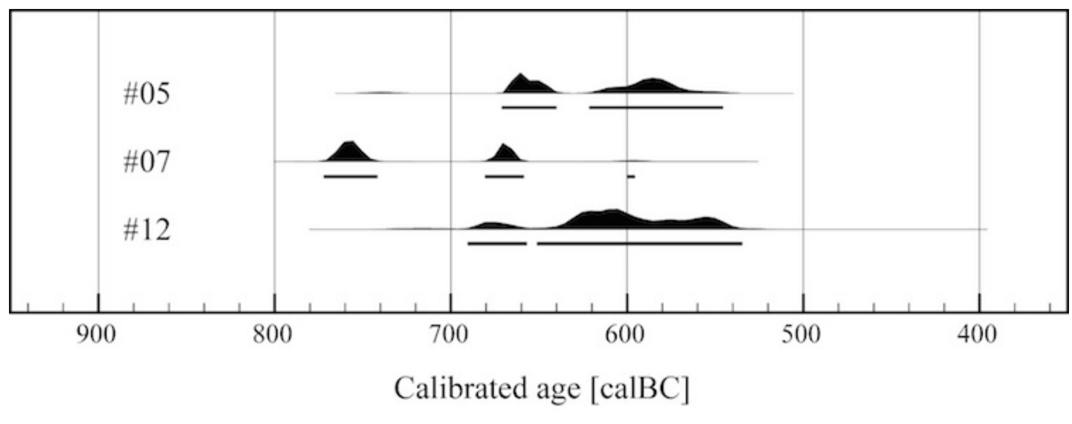

Figure 6. 2. Wiggle matching results for poles #05 (circle D or E), #07 (circle D or E), and #12 (circle E). Black lines under probability densities indicate the possible age ranges with 95.4% probability.

6. 1. 3. Circle F

Poles #04 and #06 belonging to circle F were also dated by AMS radiocarbon dating and wiggle matching. For the pole #06, we could not obtain correct radiocarbon date for one sample due to bad graphite target condition (MWK6b-50). Then it was omitted for wiggle matching analysis. All radiocarbon dates for the circle F fall in 2700-2400 BP. In this range, older dates belonging to the age range with a monotonic shape of the calibration curve mostly attribute to the pole #04. Thus, wiggle matching result for this pole introduced highly accurate date. The resulting dates for the pole #04 is 740-710 cal BC (Figure 6. 3). On the other hand, the pole #06 has more newer radiocarbon dates than the pole #04, and the wiggle matching results provided three possible age ranges from 730 to 500 cal BC (Figure 6. 3).

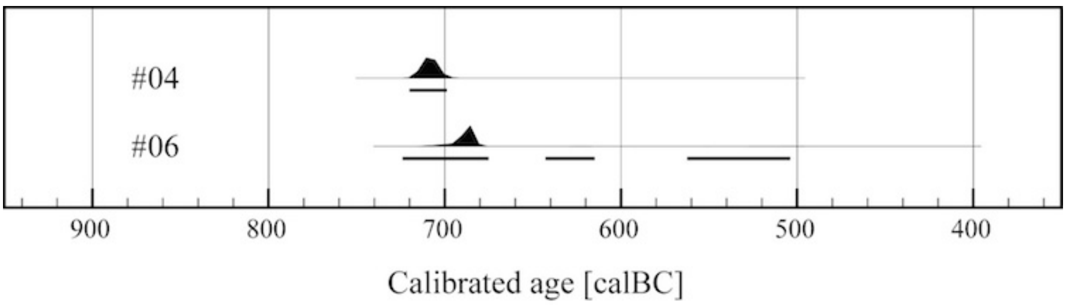

Figure 6. 3. Wiggle matching results for poles #04 and #06 (circle F). Black lines under probability densities indicate the possible age ranges with 95.4% probability.

6. 2. Conservation

There are two sections for conservation results. One is PEG elimination by chemical solvents and evaluation of the elimination using radiocarbon dating. The other is the detection test results of PEG remaining in washed conserved woods by PyGC/MS.

6. 2. 1. PEG Elimination from conserved woods

Each test sample was dated by AMS radiocarbon dating. Firstly, to confirm the contamination from PEG to woods, radiocarbon dates for pure PEG, non treated conserved woods (SS and Ar04P series), and pure woods treated with AAA (only SS series) were compared each other. The pure PEG showed radiocarbon ages of about 42,000 BP which is equivalent to the radiocarbon background of the radiocarbon dating system including sample preparation. This result provides a direct evidence that PEG is indeed manufactured from fossil fuel. Radiocarbon dates for the pure wood treated with

Table 6. 1. Radiocarbon dates for conservation materials.

Sample No.	Material	Treatment	^{14}C date [BP]	Error [1σ]	Lab. Code # [NUTA2-]
PEG7	PEG	-	42,000	230	13895
PEG8			41,700	220	13896
PEG9			41,500	220	13897
SS1-2-1	Pure Wood	AAA	4,020	30	13901
SS1-2-2			3,980	30	13904
SS1-2-3			4,010	30	13905
SS1-6-1	Conserved Wood	-	18,600	60	13909
SS1-6-2			20,000	70	13912
SS1-6-3			20,000	70	13913
Ar04P3-2-4-1	Conserved Wood	-	14,100	45	14069
Ar04P3-2-4-2			13,100	45	14070
Ar04P3-2-4-3			13,600	50	14071

Table 6. 2. Radiocarbon dates of conserved woods at the Sakuramachi site washed by AAA, AAA + hot water, AAA + acetone, or AAA + benzene, respectively

Sample No.	Material	Treatment	^{14}C date [BP]	Error [1σ]	Lab. Code # [NUTA2-]
SS1-5-1	Conserved Wood	AAA	4,200	30	13906
SS1-5-2			4,230	30	13907
SS1-5-3			4,220	30	13908
SS1-5DW-1	Conserved Wood	AAA + Hot Water	4,190	25	14251
SS1-5DW-2			4,170	25	14252
SS1-5DW-3			4,160	25	14253
SS1-7-2	Conserved Wood	AAA + Acetone	4,130	35	14079
SS1-7-3			4,170	35	14080
SS1-7-4			4,150	35	14083
SS1-4-1	Conserved Wood	AAA + Benzene	4,200	35	14076
SS1-4-2			4,240	35	14077
SS1-4-3			4,230	35	14078

Table 6. 3. Radiocarbon dates of conserved woods at the Mawaki site washed by AAA, AAA + hot water, AAA + acetone, or AAA + benzene, respectively

Sample No.	Material	Treatment	^{14}C date [BP]	Error [1σ]	Lab. Code # [NUTA2-]
Ar04P3-2-1-1	Conserved Wood	AAA	2,980	30	14059
Ar04P3-2-1-2			3,030	30	14060
Ar04P3-2-1-3			3,070	25	14061
Ar04P3-2-1DW-1	Conserved Wood	AAA + Hot Water	2,930	25	14258
Ar04P3-2-1DW-2			2,910	25	14259
Ar04P3-2-1DW-3			2,920	25	14260
Ar04P3-2-3-1	Conserved Wood	AAA + Acetone	3,040	25	14066
Ar04P3-2-3-3			3,040	30	14067
Ar04P3-2-3-4			3,100	25	14068
Ar04P3-2-2-1	Conserved Wood	AAA + Benzene	3,140	30	14062
Ar04P3-2-2-3			3,080	25	14063
Ar04P3-2-2-4			3,030	25	14065

AAA to be assumed at the true value for SS series were almost 4,000 BP, but the SS series woods conserved with PEG were dated more older as *ca.* 20,000 BP. Therefore, it was clearly revealed that dead carbon originated from PEG disturbed accurate radiocarbon dating.

In addition, for Ar04P series, true age estimated from dendrochronological cross-dating is around 2,410 BP, radiocarbon dates of the conserved woods were determined also to be older than the true ages, as *ca.* 13,600 BP. As the results, we conclude that PEG is actually made from fossil fuel and conserved woods with PEG are contaminated by dead carbon originated from fossil fuel.

In the next, we also compared efficiency of PEG elimination by four treatment methods (Table 6. 2. and 6. 3.). Conserved woods of SS and Ar04P series treated only the AAA treatment obtained radiocarbon dates of ca 4,030 and 3,000 BP, respectively. These dates are clearly older than true age and indicate existence of the PEG effect even after the AAA treatment. Therefore, it was revealed that the AAA treatment can not remove PEG completely from the conserved woods. As additional treatments,

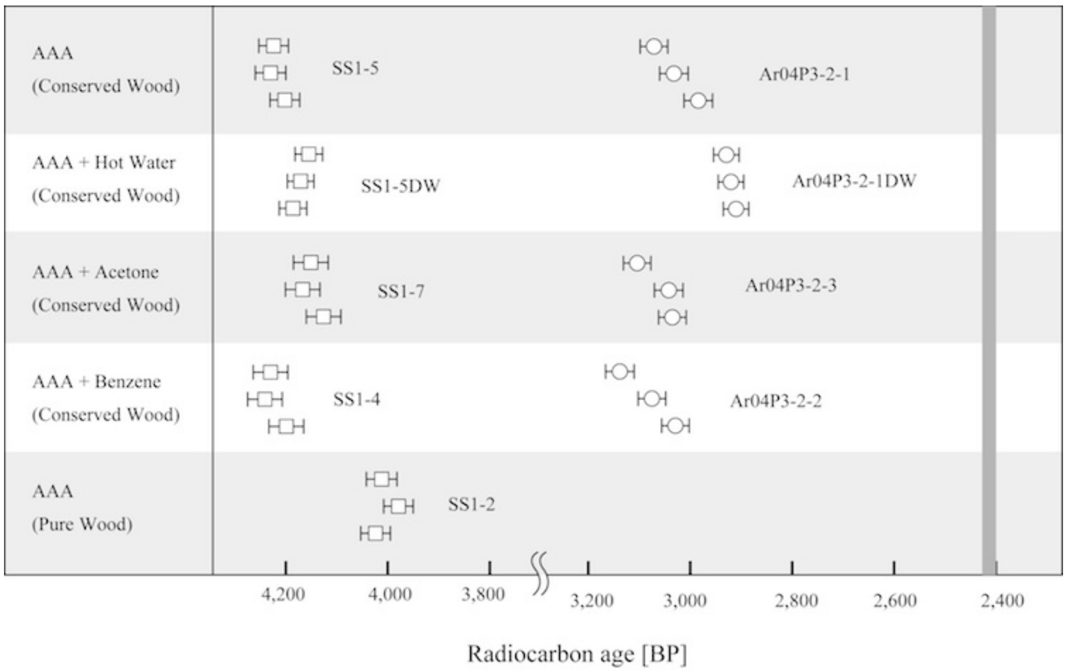

Figure 6. 4. Comparison of radiocarbon ages of the woods conserved with PEG washed by different washing processes.

conserved woods were washed by hot water, acetone, and benzene after the AAA treatment. Hot water samples were dated as *ca.* 4,170 and 2,920 BP, respectively.

6. 2. 2. PEG detection by GC/MS

In order to detect PEG remaining in conserved waterlogged wood, we firstly measured PyGC/MS of pure PEG and pure wood. Obtained chromatograms from the pure PEG, three characteristic peaks were periodically observed in total ion chromatogram and named as A_n, B_n, and C_n, respectively (Figure 6. 5). In contrast, on chromatograms from the pure wood, only A_n, and B_n were observed (Figure 6. 6). The results mean that the PEG may be characterized by the peak C_n. To identify the peak C_n, MS of molecular ions and fragment ions were qualitatively measured for each peak. Consequently, C_n series was identified as fragments of PEG (Figure 6. 7). We also analyzed conserved woods treated with AAA (still remaining PEG). As the result, the C_n was detected from the conserved wood, then existence of PEG in the conserved was detected from PyGC/MS.

7. Discussion

7. 1. Chronology of wooden circular structures

Wiggle matching results introduced highly accurate chronology for wooden circular structures. Using these results and also chronological studies such as cross-dating and single radiocarbon dating reported from previous studies, we discussed construction period of the circular structures at the Mawaki site.

7. 1. 1. Construction period of circle A

Wiggle matching results introduced precise dates for poles #01, #02, #03, #15 belonging to circle A. However only the pole #03 has older dates (950-840 cal BC) than other poles (approximately 800 cal BC). These resulting dates were compared with wiggle matching results of a cedar plate which was excavated under the pole #02 and dated by Nakamura *et al*. (2007). The dates of cedar plate (810-780 cal BC) is consistent with our results of poles #01, #02, and #15. Therefore, valid construction period of the circle A is estimated as around 800 cal BC. The older date of the pole #03 may sugest that the wood was cut a few tens of years in advance for the construction of circle A.

7. 1. 2. Construction period of circle D, E

Wiggle matching results for poles #05, #07, and #12 forming circle D and/or E suggest that their felling dates were widely spreading in the age range from 770 to 540 cal BC. Because each pole has two or three possible age ranges with 95.4% probability on the calibration curve, it is difficult to estimate more precise dates for the poles, and we concluded that the construction period of the circle D and E is approximately 770-540 cal BC.

7. 1. 3. Construction period of circle F

Wooden poles #04 and #06 from circle F were dated by wiggle matching. The dates distributed widely from 740 to 500 cal BC. The pole #06 has three possible age ranges (730-680, 640-610, 560-500 cal BC) in 95.4% probability on the calibration curve from 730 to 500 cal BC. On the other hand, the pole #04 is fortunately dated precisely from 740 to 710 cal BC. When we assume that dates of two poles belonging to the same circle should be contemporaneous, newer dates (640-610 and 560-500 cal BC) of the pole #06, which are not consistent with the dates of the pole #04, can be omitted. Therefore, the construction period of the circle F is estimated as 740-680 cal BC, which is the age range overlapped by the estimates from the poles #04 and #06.

7. 1. 4. Construction period of wooden circular structures

Main subject of this study is to determine the construction period of the wooden circular structures at the Mawaki site. As mentioned above, we decided the construction period of the circle A, D/E, F. Totally, time interval of the construction periods was distributed from 820 to 540 cal BC. However this was estimated without dates of circle B and C. To determine more reliable construction period, dates for the both circles B and C are required. Although we did not measure radiocarbon dates for the circle B and C, because of carbon contamination from PEG, Yamada (1986) reported a radiocarbon date of pole #24 (circle B). The calibrated ages are 890-875 and 850-790 cal BC, and the oldest age for all poles excavated at the Mawaki site is obtained from the pole #24. Consequently, it was revealed that the construction of the wooden circular structures was lasted from 890 to 540 cal BC at the Mawaki site.

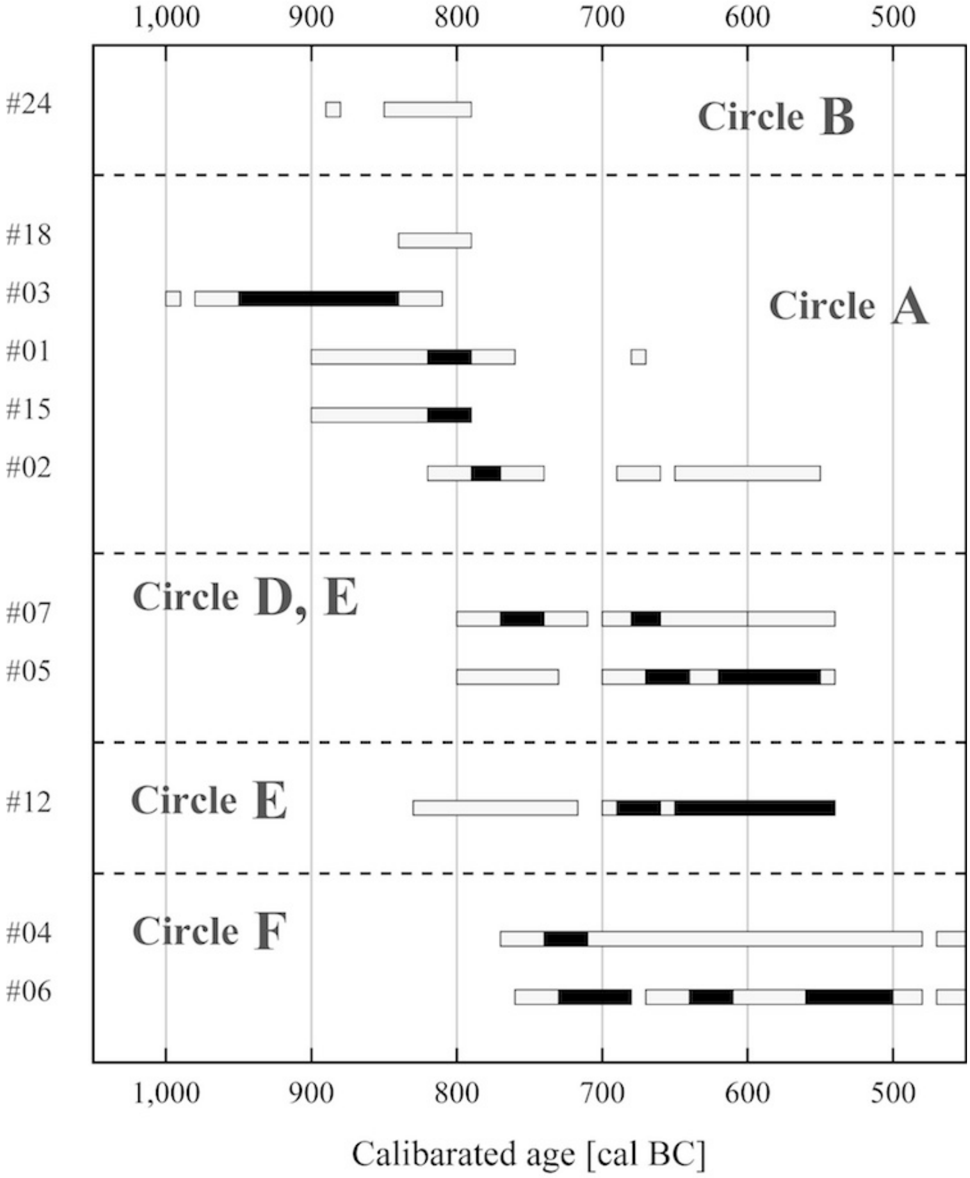

Figure 7. 1. Calibrated ages of wooden poles forming circular structures at the Mawaki site. Closed bars indicate wiggle matching results in 95.4% probability density. Open bars were calibrated dates (95.4%) of the outermost ring of each pole. Radiocarbon dates for poles #24 and #18 were measured by Yamada (1986).

7. 1. 5. Chronology on the Final Jomon period

From the archaeological studies, such as pottery-type analysis, the Final Jomon period is divided to the first half (*ca.* 1,300-900 cal BC) and the last half (*ca.* 900-500 cal BC). In the coastal region around Noto Peninsula, diminution in the number of settlements, *i.e.* the weakening of human activity, from the first half to the last half is recognized archaeologically. On the other hand, the construction period of the wooden circular structures at the Mawaki site was estimated to be 890-540 cal BC with the wiggle matching method, and this period belongs to the last half of the Final Jomon period. This result implies that the wooden circular structures were constructed even under the environment of decreasing human population. However, at the Sakuramachi site, the ages of the wooden circular structures (*ca.* 1125-845 cal BC) coincide with intermediate period between the first half and the last half of the Final Jomon period. In addition, the time difference was observed between at the Mawaki site and the Sakuramachi site. Thus it demonstrates that the construction of the wooden circular structures was started from the intermediate time and lasted during the last half of the Final Jomon period.

7. 2. Conserved waterlogged woods with PEG

In order to eliminate PEG from waterlogged woods, four washing methods (AAA, AAA + D. W., AAA + acetone, AAA + benzene) was examined. From the results, possibility of PEG elimination was discussed. In addition, the most effective method for the elimination is selected provisionally.

As measurement tools of PEG remaining in the waterlogged woods, GC/MS of the wood were also tried. Effectiveness of PEG detection was considered.

7. 2. 1. Possibility of PEG elimination

Here we discuss PEG elimination effect among four washing methods. For SS series woods which were soaked into PEG (PEG4000s made by Sanyo Chemical

Industries, Ltd.) for this study, age differences between radiocarbon dates of washed samples and pure woods treated by AAA (*ca.* 4000 BP means true radiocarbon date) indicated that the most effective method is AAA + hot water or AAA + acetone. Although absolute elimination of PEG could not be achieved even these methods, this result implies that when we continue washing processes with hot water or acetone after AAA, PEG will be removed completely.

Ar04P series were conserved in 1980s with PEG, we could not obtain non-processed wood which bring us true radiocarbon dates. Then the true dates were estimated from dendrochronological cross-dating. The estimated true radiocarbon date of the Ar04P series (approximately 2400 BP) were compared with measured radiocarbon dates of wood samples washed by four methods. As the result, all radiocarbon dates of the washed samples were older than the estimated true date, and it is revealed that PEG is still remaining in the washed samples. In whole samples, the nearest radiocarbon dates to the true value were obtained from samples treated with AAA + hot water. This method demonstrated good result for SS series, as well. Hence, we conclude that the most appropriate and effective elimination method of PEG from conserved waterlogged wood is the AAA + hot water process.

7. 2. 2. PEG detection using GC/MS

Comparing with chromatograms measured by PyGC/MS between pure PEG and pure wood, it was suggested that characteristic peak series (C_n) in the PEG will be utilized to detect PEG remaining in wood conserved with PEG. Actually, Cn was observed on chromatograms of conserved woods contain 4% PEG, detection of PEG using PyGC/MS was achieved successfully. In addition, from mass spectra of Cn, fragments of PEG were apparently identified. Thus, Cn series observed on chromatograms of the conserved woods is some fragments of PEG, PyGC/MS has ability to detect PEG from wood absolutely. For the PyGC/MS, we required only *ca.* 5mg unwatered wood samples. Therefore, PyGC/MS is also practical analytical method to measure conserved waterlogged woods.

8. Conclusion

8. 1. Wooden circular structures

In order to introduce precise construction period of the wooden circular structures detected from the Mawaki site, wiggle matching analysis was applied for annual ring sequences taken from chestnut timbers constituting the structures. Wiggle matching results revealed that the construction period of the circular structures at the Mawaki site is *ca.* 890-540 cal BC. This period corresponds to the last half of the Final Jomon period. From archaeological research on typology of pottery and counting of settlement sites, it is regarded that human activity weakened in the last half of the Final Jomon period. Therefore, it can be suggested that there is some sort of a relationship between the construction of the wooden circular structures and the weakening of human activity during the last half of the Final Jomon period.

The estimated construction period was compared with the dates of circular structures excavated at the Sakuramachi site (1125-845 cal BC). Time difference was observed clearly, and the structures at the Sakuramachi site have older dates than the construction period of the Mawaki site. The dates of the Sakuramachi site relate with the intermediate period of the Final jomon period, therefore, it can be presumed that the wooden circular structures had been constructed from the intermediate period to the last half of the Final Jomon period at the coastal region around Noto Peninsula.

8. 2. Elimination and detection of PEG

We tried elimination of PEG from conserved waterlogged woods using not only AAA treatment, but also additional processing (*i.e.,* washing by hot water, acetone, benzene). To estimate degree of the elimination, the washed samples were dated by AMS radiocarbon dating and compared with true radiocarbon ages of the sample. As the results, all washed samples obtained contaminated radiocarbon dates, *i.e*., the radiocarbon dates assigned systematically to older dates than the true ages due to dead carbon from remaining PEG in the washed samples. This result revealed that AAA treatment and the additional processing cannot eliminate PEG from conserved wood completely, and is inconsistent with a conclusion by Bruhn *et al.* (2001). Between this study and the previous study, there are several differences of experimental condition such as molecular weight of PEG used for impregnation, soaking time of PEG solution into waterlogged wood, and species of the waterlogged wood. Therefore, it was suggested that possibility of the PEG elimination is deeply dependent on the experimental condition. We were also compared radiocarbon dates between the washed samples. Consequently, washing by AAA + hot water is regarded as the most effective treatment process because treated samples by this method have close radiocarbon dates to the true date.

On the other hand, in order to detect PEG surviving in the conserved woods, PyGC/MS of PEG and the conserved woods were performed. Then characteristic chromatograms which indicate existence of PEG were observed by PyGC/MS. Hence PyGC/MS can be used as a detector of PEG remaining in the conserved woods. As further approach, it is anticipated that quantitative research of the remaining PEG using standard curve built from GC of known concentration PEG. As stated above, although complete elimination of PEG was impossible in our methods, radiocarbon dates contaminated from remaining PEG may be corrected by PEG amount estimated from GC.

Acknowledgments

I am deeply grateful to My supervisor Professor Toshio Nakamura for helpful advice and great support throughout my research. Professor Kazuhiro Suzuki and Dr. Masayo Minami have given me invaluable advices and assistance. Dr. Hirotaka Oda introduced me the principal methodology of radiocarbon dating. Ms Tomoko Ohta and Miss Akiko Ikeda provided me the procedure of sample preparation, and Mr Takayuki Omori assisted statistic analyses in this work, and this is greatly appreciated.

I would like to Professor Katsuhiko Kimura of Fukushima University, Japan, for dendrochronological analysis of the wooden poles, and Mr. Shinya Nakamura of Kanazawa Gakuin University, for processing of the conservation with PEG.

This work was supported partly by the 'Grant-In Aid for Scientific Research' of the Japan Society for the Promotion of Science (JSPS) (subject #19300300, 16320108).

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Appendix I

Results of radiocarbon dating by this experiment

Table A.I

Radiocarbon ages of tree ring sequence collected from pole #01 which belongs to the circle A

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #01 (A)	MWK1a-4-1	-26	2624	41	12337
	MWK1a-14	-26	2655	41	12338
	MWK1a-28	-26	2685	41	12339
	MWK1a-46	-26	2708	41	12340
	MWK1a-58	-24	2710	41	12341
	MWK1a-63	-25	2756	41	12342
	MWK1a-76	-25	2735	41	12345
	MWK1a-88	-23	2675	41	12346
	MWK1a-94	-26	2687	41	12347
	MWK1a-105	-24	2816	41	12348
	MWK1a-122	-25	2795	41	12350

Table A.II

Radiocarbon ages of tree ring sequence collected from pole #02 which belongs to the circle A.

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #02 (A)	MWK2-3	-29	2575	40	12351
	MWK2-8	-28	2535	41	12354
	MWK2-12	-26	2578	41	12355
	MWK2-18	-26	2615	41	12356
	MWK2-22	-28	2591	41	12357
	MWK2-26	-26	2671	41	12358
	MWK2-34	-28	2665	41	12359
	MWK2-39	-28	2643	41	12361
	MWK2-44	-28	2681	41	12362
	MWK2-48	-26	2693	41	12349

Table A. III

Radiocarbon ages of tree ring sequence collected from pole #03 which belongs to the circle A.

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #03 (A)	MWK3a-4	-26	2735	46	15013
	MWK3a-28	-26	2786	46	15014
	MWK3a-50	-27	2753	46	15015
	MWK3a-72	-24	2842	46	15016
	MWK3a-94	-25	2839	46	15017
	MWK3a-116	-31	2880	50	15018

Table A.IV

Radiocarbon ages of tree ring sequence collected from pole #15 which belongs to the circle A

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #15 (A)	MWK15-1	-24	2667	34	13056
	MWK15-3	-26	2623	33	13057
	MWK15-6	-26	2633	33	13058
	MWK15-14	-29	2631	33	13060
	MWK15-22	-26	2619	34	13061
	MWK15-26	-27	2694	33	13062
	MWK15-28	-27	2699	37	13063
	MWK15-32	-26	2743	33	13064

Table A.V

Radiocarbon ages of tree ring sequence collected from pole #05 which belongs to the circle D and E.

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #05 (D, E)	MWK5P-1	-30	2539	34	14522
	MWK5P-2	-28	2457	32	14523
	MWK5-3	-29	2475	32	14524
	MWK5-6	-29	2476	32	14525
	MWK5-10	-28	2533	32	14526
	MWK5-14	-28	2512	32	14527
	MWK5-18	-27	2548	32	14529
	MWK5-22	-25	2545	33	14530
	MWK5-26	-28	2534	32	14531
	MWK5-30	-27	2482	32	14532
	MWK5-34	-26	2605	32	14533
	MWK5-37	-26	2540	31	14534
	MWK5-38	-27	2465	31	14535
	MWK5-39	-26	2437	31	14536

Table A.VI

Radiocarbon ages of tree ring sequence collected from pole #07 which belongs to the circle D and E.

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #05（D, E）	MWK7-1	-25	2518	31	14538
	MWK7-2	-25	2502	31	14539
	MWK7-4	-26	2538	31	14540
	MWK7-6	-25	2514	31	14541
	MWK7-8	-26	2540	32	14542
	MWK7-10	-25	2549	31	14543
	MWK7-12	-26	2555	32	14544
	MWK7-14	-24	2528	31	14546
	MWK7-16	-25	2523	32	14547
	MWK7-18	-26	2573	32	14548
	MWK7-20	-26	2500	32	14549
	MWK7-22	-28	2540	32	14550
	MWK7-23	-29	2499	32	14551

Table A.VI

Radiocarbon ages of tree ring sequence collected from pole #07 which belongs to the circle E.

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #12 (E)	MWK12b-2	-29	2577	48	15020
	MWK12b-4	-28	2492	46	15021
	MWK12b-7	-27	2458	46	15022
	MWK12b-11	-27	2503	47	15023
	MWK12b-15	-27	2501	46	15024
	MWK12b-19	-27	2478	46	15025
	MWK12b-23	-25	2503	46	15026
	MWK12b-27	-25	2496	46	15027
	MWK12b-31	-26	2456	46	15029
	MWK12b-35	-27	2460	46	15030
	MWK12b-39	-26	2495	46	15031
	MWK12a-39	-26	2472	46	15032

Table A.VII

Radiocarbon ages of tree tree ring sequence collected from pole #04 which belongs to the circle F.

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #04（F）	MWK4b-4	-26	2475	33	13027
	MWK4b-12	-25	2435	32	13028
	MWK4b-19	-25	2481	33	13029
	MWK4b-26	-27	2516	33	13030
	MWK4b-36	-25	2507	33	13031
	MWK4b-40	-25	2511	33	13032
	MWK4b-46	-25	2529	33	13034
	MWK4b-53	-25	2519	33	13035
	MWK4b-59	-24	2543	33	13036
	MWK4b-67	-26	2539	33	13037
	MWK4b-72	-27	2550	33	13038
	MWK4b-79	-27	2625	33	13039
	MWK4b-87	-29	2651	34	13040

Table A.VII

Radiocarbon ages of tree ring sequence collected from pole #06 which belongs to the circle F.

Poles No. (Circle)	Sample No.	$\delta^{13}C$ (‰)	^{14}C age (BP)	Error (±1σ)	Lab Code # (NUTA2-)
Poles #06 (F)	MWK6b-4	-25	2472	33	13041
	MWK6b-6	-26	2476	33	13043
	MWK6b-10	-27	2458	33	13044
	MWK6b-15	-26	2492	33	13045
	MWK6b-18	-26	2474	33	13046
	MWK6b-23	-26	2463	33	13047
	MWK6b-26	-25	2432	33	13048
	MWK6b-29	-25	2471	33	13049
	MWK6b-33	-26	2417	32	13051
	MWK6b-36	-25	2473	32	13052
	MWK6b-39	-24	2450	32	13053
	MWK6b-44	-24	2494	33	13054
	MWK6b-50	-32	2648	46	13055

Appendix II

Results of wiggle matching

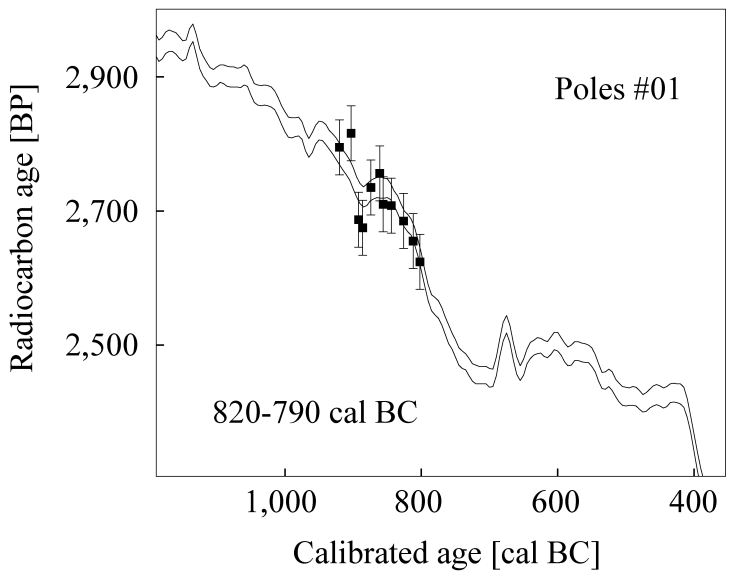

Figure A.I. Wiggle matching result of poles #01 (circle A). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

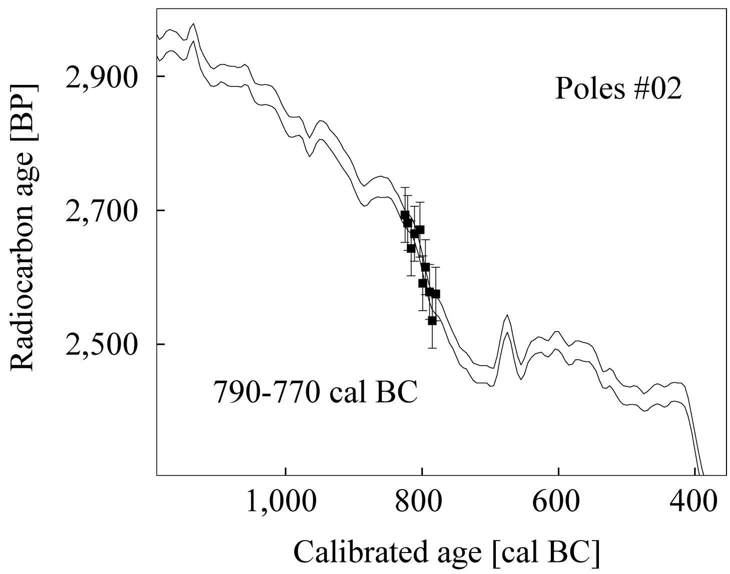

Figure A.II. Wiggle matching result of poles #01 (circle A). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

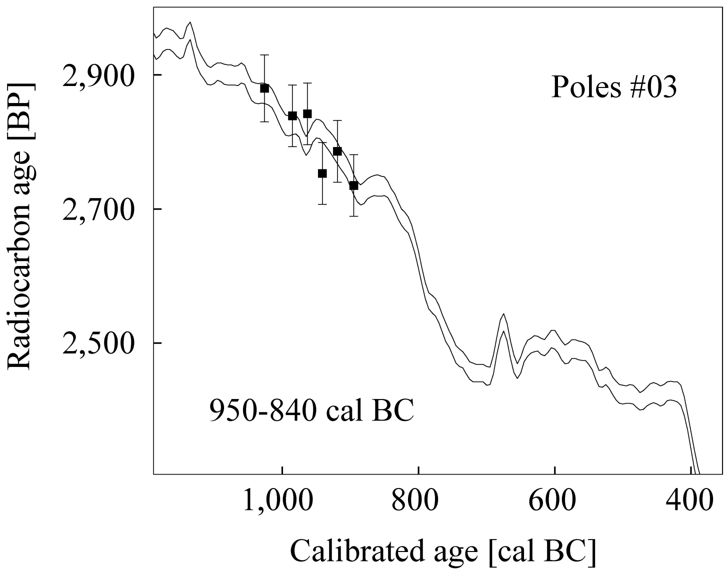

Figure A.III. Wiggle matching result of poles #03 (circle A). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

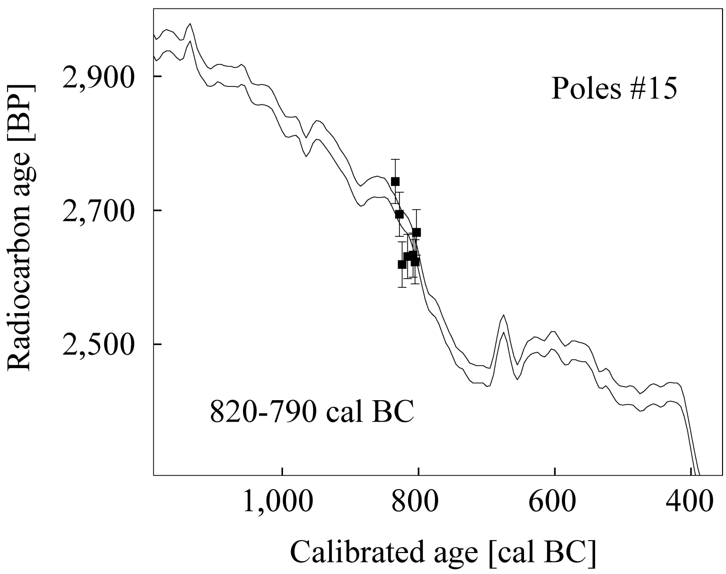

Figure A.IV. Wiggle matching result of poles #15 (circle A). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

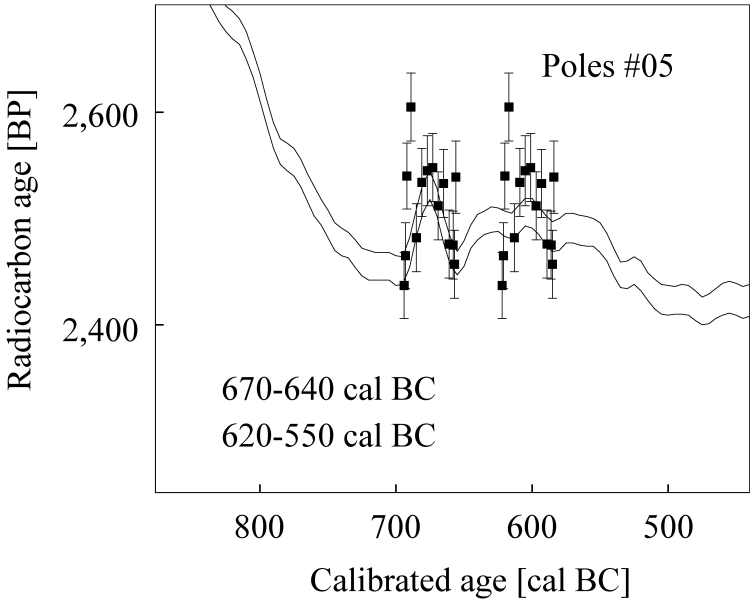

Figure A.V. Wiggle matching result of poles #05 (circle D, E). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

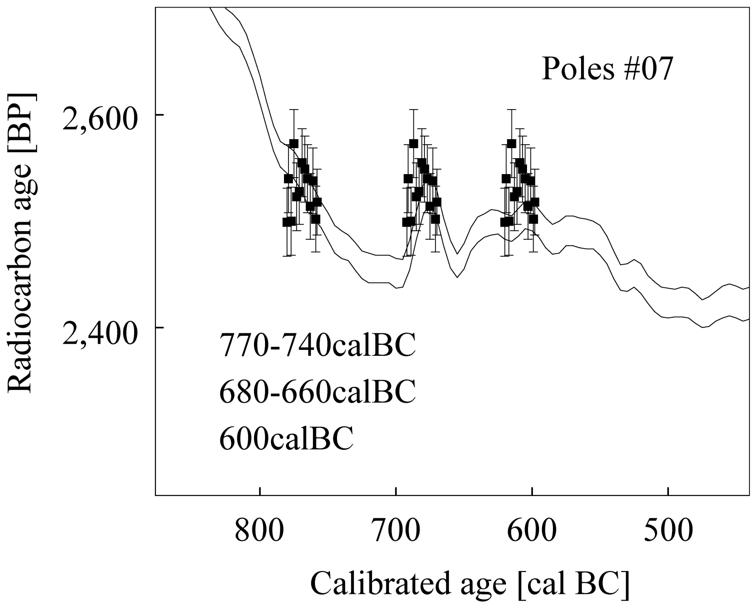

Figure A.VI. Wiggle matching result of poles #07 (circle A). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

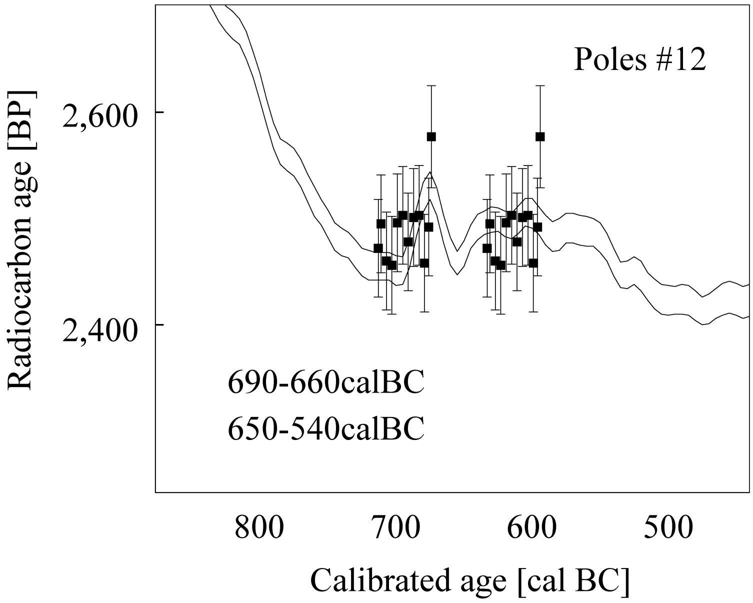

Figure A.VII. Wiggle matching result of poles #12 (circle E). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

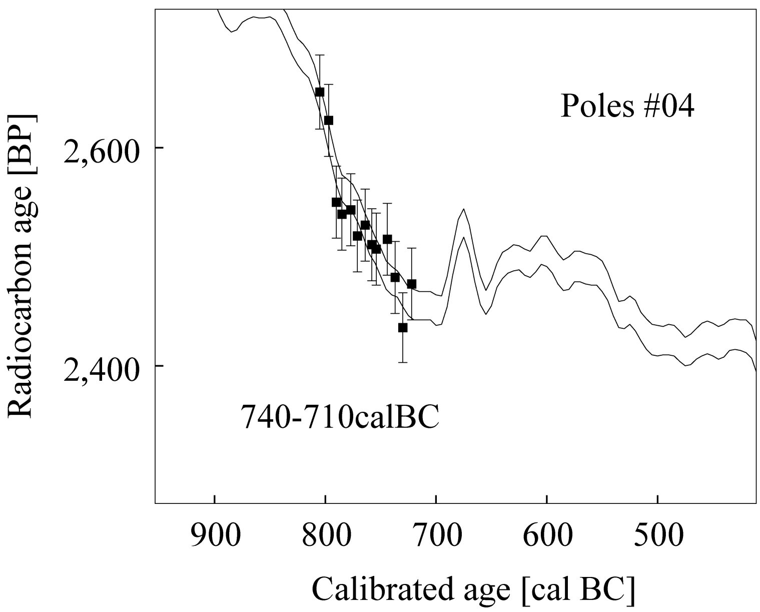

Figure A.VIII. Wiggle matching result of poles #04 (circle F). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

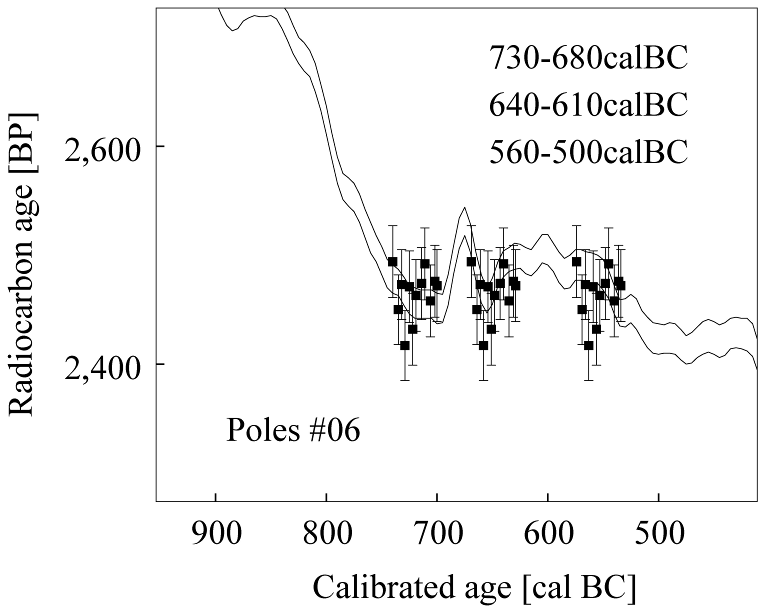

Figure A.IX. Wiggle matching result of poles #06 (circle F). Sequencial radiocarbon dates were plotted on the calibration curve IntCal09.

学 位 審 査 論 文 目 録

報告番号	※ 第 号	氏 名	西本 寛

主 論 文
題 目

High precision radiocarbon dating of archaeological waterlogged wood: focusing on wooden poles forming circular structures at the Mawaki site

（既に印刷公表したものについては、その方法および年月日、未公表のものについては、公表の方法および時期を記入すること。）

副 論 文
題 目

Nishimoto H., Nakamura T., Takada H. 2010, Radiocarbon dating and wiggle matching of wooden poles forming circular structures in the 1st Millennium BC at the Mawaki archaeological site, central Japan. *Nucl. Instrum. Meth. B* vol. 268, 1026-1029.

（ 同 上 ）

参考論文
題 目

Nishimoto H., Takada H., Nakamura T., 2010, Precise dating of waterlogged woods forming circular structures at the Mawaki site, central Japan, by wiggle-matching analysis, *Proceedings of the International Workshop on Low-Level Measurement of Radionuclides and Its Application to Earth and Environmental Sciences*, 75-80.

（ 同 上 ）